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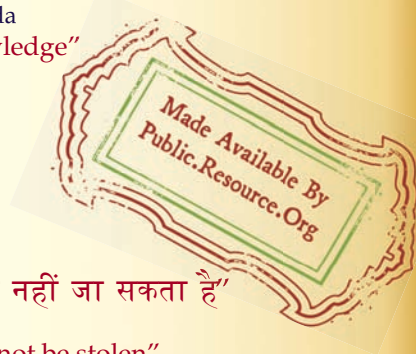
IS 14162 (1994): Determination of fosetylAl residues in agricultural and food commodities [FAD 1: Pesticides and Pesticides Residue Analysis]



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“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

कीटनाशी — कृषि, खाद्य वस्तुओं, मिट्टी एवं
पानी में फासटिल ज्ञात करने की पद्धति

Indian Standard

**PESTICIDE — DETERMINATION
OF FOSETYL-AL RESIDUES IN
AGRICULTURAL AND FOOD
COMMODITIES**

UDC 664 : 543.544.25 [632.952.28 FOS]

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Price Group 2

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Fosetyl-Al formulations are used as fungicides in agriculture, specially in-horticulture. This standard will enable the food, health authorities and others engaged in the field to follow uniform test procedure for the estimation of residues of Fosetyl-Al in various agricultural and food commodities.

In the preparation of this standard, due consideration has been given to the provisions of ***Prevention of Food Adulteration Act, 1954*** and Rules framed thereunder and ***Standards of Weights and Measures (Packaged Commodities) Rules, 1977***.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (***revised***)'.

Indian Standard

PESTICIDE — DETERMINATION OF FOSETYL-AL RESIDUES IN AGRICULTURAL AND FOOD COMMODITIES

1 SCOPE

1.1 This standard prescribes a gas chromatographic method for determination of residues of fosetyl-Al [aluminium tris (ethyl phosphonate)] and its major metabolite, phosphonic acid in agricultural and food commodities.

1.2 This method has a detection limit lower than of $0.05 \mu\text{g/g}$ (0.05 ppm) using a phosphorus specific flame-thermionic detector.

2 REFERENCES

The Indian Standards listed at Annex A are necessary adjuncts to this standard.

3 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1992) shall be employed in tests.

NOTE 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4 SAMPLING

The representative samples for the purpose of estimating fosetyl-Al residues in the samples shall be drawn in accordance with IS 11380 : 1985.

5 PRINCIPLE

Ethylphosphonic acid (I) (the anion of fosetyl-Al) and its major metabolite, phosphonic acid (II) are extracted from the sample by maceration with 0.1 M tartaric acid. The aqueous extract is added to propan-2-ol and the mixture is then methylated with an ethereal solution of diazomethane. Ethyl methyl phosphonate and dimethyl phosphonate formed by methylation of (I) and (II) respectively are determined by gas chromatography using a phosphorus specific flame-thermionic detector.

6 'SAMPLE STORAGE

Sample shall be stored as such when chances of its degradation do not exist and the quantity is small. Otherwise extraction shall be carried out and the extract stored. Depending upon the nature of the sample, keep the sample or its extract either in

deep-freezer at -15°C or in refrigerator until taken up for analysis. Ensure that the samples do not absorb or loose moisture during storage. Avoid undue long storage period.

7 APPARATUS

7.1 Gas Chromatograph

The gas chromatograph shall be fitted with a phosphorus specific flame — thermoionic detector and printer — plotter cum integrator. The following operating parameters are suggested, which can be changed provided standardization is done:

Column: Glass, 200 cm length x 3 mm I.D.; packed with Chromosorb Century 101 (80-100 mesh)

Temperature:

Column oven	180°C
Injection port	200°C
Detector	200°C

Carrier Gas and Flow Rate: Helium; 60 ml/min

Alternative Gas Chromatographic Conditions

Column: Glass, 200cm length x 3 mm I.D.; packed with 1.7 m Carbowax 20 M 20 percent on Chromosorb WHP (SO-100 mesh) with 0.3 m Chromosorb 101 (100-120 mesh)

Temperature:

Column oven	170°C
Injection port	200°C
Detector	200°C

Carrier Gas and Flow Rate: Nitrogen; 60 ml/min

7.1.1 **Microtitre Syringe** — 10 μl capacity.

7.1.2 **Waring Blender or Equivalent**

7.1.3 **Centrifuge**

7.1.4 **Rotary Evaporative Concentrator**

7.1.5 **Sample Grinder**

7.1.6 **Mechanical Shaker**

7.1.7 **Ultrasonic Bath**

7.1.8 **Chromatographic Column** — glass, 200 cm x 18 mm I.D. with ground glass top.

7.1.9 Fume Cupboard

7.2 Reagents

7.2.1 *Propan-2-ol* — glass re-distilled.

7.2.2 *Aqueous Tartaric Acid* — 0.1 M.

7.2.3 *Aqueous Tartaric Acid* — 1 M

7.2.4 *Reference Standard Fosetyl-Al* — of known purity.

7.2.5 *Reference Standard Sodium Phosphite Acid* — of known purity.

7.2.6 *Diethyl Ether* — glass re-distilled.

7.2.7 *Methylating Reagent (Diazomethane)* — As given in Annex B.

7.2.8 *Ethanol* — glass re-distilled.

7.2.9 *Potassium Hydroxide Solution* — 37.5 percent solution in water (*m/v*).

7.2.10 *N-methyl-N-nitrosotoluene-4-sulphonamide Solution* — 20 percent solution in diethyl ether (*m/v*).

7.2.11 *Glacial Acetic Acid* *

8 EXTRACTION

Immediately prior to analysis, blend the frozen crop samples using cutter/blender to make the mass homogeneous. Take care not to allow the sample to thaw. Weigh about 25 g of the homogeneous sample into a 100-ml centrifuge tube. Add 40 ml 0.1 M tartaric acid and macerate for 2 minutes at high speed. Wash the macerator with about 5 ml distilled water. Centrifuge the sample for 10 minutes at 4 000 rotations per min. Remove the supernatant liquid and transfer it to a 100-ml volumetric flask. Add 40 ml 0.1 M tartaric acid to the sample and macerate. This volume of tartaric acid is adjusted, so that final extract makes the final volume of extract as near to 100 ml. Wash the macerator using approximately 5 ml of water, adding the washings to the sample. Centrifuge as before. Remove the supernatant liquid and transfer it to the volumetric flask containing the initial extract. Make up the volume to the mark with distilled water.

9 PREPARATION OF STANDARD AND SAMPLE SOLUTIONS

9.1 Fosetyl-Al Standard Solution

Weigh accurately about 0.1 g of reference standard into a 100-ml volumetric flask and make up the volume with water at 20°C and shake to mix well.

9.2 Phosphonic Acid Standard Solution

Weigh accurately 0.263 4 g of sodium phosphite (equivalent to 0.1 g of phosphonic acid) into a

100-ml volumetric flask and dissolve in water at 20°C. Make up the volume to the mark.

9.3 Mix Standard Solution

Mix about 1 ml each of standard solutions as at 9.1 and 9.2.

9.4 Methylator

9.4.1 Standard Solution

Take an aliquot of 1 ml of the aqueous standard solution mixture (9.3) into a 10-ml volumetric flask and add 5 ml propan-2-ol and 2 drops of 1 M aqueous tartaric acid. Add to this mixture, sufficient methylating reagent to produce a permanent pale yellow colour. Allow this solution to stand for 1 minute and then destroy the excess of methylating reagent by dropwise addition of 1 M aqueous tartaric acid. Remove the diethyl ether present with a stream of nitrogen flowing at the rate of 0.5 l/min. Then make up the volume to the mark with propan-2-ol.

9.4.2 Extract Solution

Take an aliquot of 1 ml of the aqueous extract (8) into a 10-ml volumetric flask and add 5 ml propan-2-ol and 2 drops of 1 M aqueous tartaric acid. Add to this mixture, sufficient methylating reagent to produce a permanent pale yellow colour. Allow this solution to stand for 1 minute and then destroy the excess of methylating reagent by dropwise addition of 1 M aqueous tartaric acid. Remove the diethyl ether present with a stream of nitrogen flowing at the rate of 0.5 l/min. Then make up the volume to the mark with propan-2-ol.

9.5 Standard Solution for Gas Chromatography

Serially dilute accurately measured volumes of the mixed standard solution (9.4.L) with 10 percent (v/v) of distilled water in propan-2-ol at 20°C to give solutions of a useful range for the samples and recoveries that are quantified. Store the aqueous standard solution at 4°C for up to 48 hours. Store methylated standards at -20°C for up to 1 week.

10 ESTIMATION

10.1 Inject into the gas chromatograph, with the help of a microlitre syringe, a suitable aliquot of the methylated mixed standard solution and measure the peak areas. Similarly inject into the gas chromatograph, with the help of a microlitre syringe, a suitable aliquot of the methylated sample extract, identify the peaks by the retention times and measure the peak areas. Estimate the fosetyl-Al content by comparison with the standard curve.

11 CALCULATION

$$\text{Fosetyl-Al residue, } \mu\text{g/g} = \frac{A_1 \times V_2 \times V_3 \times C}{A_2 \times V_1 \times M} \times f$$

where

- A_1 = peak area of the sample;
 V_2 = volume, in μl , of methylated mixed standard solution injected;
 V_3 = total volume, in ml, of sample solution;
 C = concentration, in $\mu\text{g/g}$, of the methylated mixed standard solution;

$$f = \text{recovery factor} = \frac{100}{\text{percent mean recovery}};$$

A_2 = peak area of the methylated mixed standard solution;

V_1 = volume, in μl , of sample solution injected; and

M = mass, in g, of sample taken for analysis.

NOTE — Percent mean recovery is determined by taking untreated control sample to which known amounts of fosetyl-Al and phosphonic acid standards are added and analysed as described above.

ANNEX A

(Clause 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	Title	IS No.	Title
1070 : 1992	Reagent grade, water (third revision)	11380 : 1985	Methods of sampling for determination of pesticide residues in agricultural and food commodities

ANNEX B

(Clause 7.2.7)

PREPARATION OF METHYLATING REAGENT (DIAZOMETHANE)**B-1 METHOD**

Diazomethane is prepared using a specially designed generator. Warm up the water-bath to 50°C. Charge the apparatus with 50 ml potassium hydroxide solution. Wash the potassium hydroxide solution with 30 ml ethanol. Add under nitrogen, 50 ml of N-methyl-N-nitrosotoluene-4-sulphonamide solution. Collect the diazomethane evolved in 20 ml diethyl ether.

B-2 PRECAUTIONS

B-2.1 Both diazomethane and N-methyl-N-nitrosotoluene-4-sulphonamide are carcinogenic. Therefore, before preparing or using diazomethane, follow these instructions.

B-2.1.1 Diazomethane must not be prepared by inexperienced personnel without adequate supervision.

11-2.1.2 Prepare diazomethane only in a fume cupboard with the front door closed, as much as possible.

B-2.1.3 Display the appropriate notices on the laboratory doors and on the fume cupboard front. Restrict access to the laboratory from the corridor.

B-2.1.4 Always wear gloves. Dispose of these in a suitable container which is to be sealed before it is removed from the fume cupboard.

B-2.1.5 Always wash the glassware used with copious amounts of water and ethanol before removing it from the fume cupboard. Dispose of the solvent in a suitable, labelled container.

B-2.1.6 Destroy any excess diazomethane by the dropwise addition of glacial acetic acid.

B-2.1.7 Prepare only sufficient diazomethane for your immediate needs.

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